

S/N 09/614,631

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicant: HILGREN ET AL. Examiner: J. PAK
Serial No.: 09/614,631 Group Art Unit: 1653
Filed: JULY 12, 2000 Docket No.: 163.1382US01
Title: METHOD AND COMPOSITION FOR INHIBITION OF MICROBIAL
GROWTH IN AQUEOUS FOOD TRANSPORT AND PROCESS
STREAMS

CERTIFICATE UNDER 37 CFR 1.6(d):

I hereby certify that this paper is being transmitted by facsimile to the U.S. Patent and Trademark Office on January 27, 2006.

By: 

Name: Kay Fahland

DECLARATION UNDER 37 CFR § 1.131

Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

I, John D. Hilgren, declare and state the following:

1. I am an inventor of the subject matter of the patent application identified above and an employee of Ecolab, Inc., the assignee of the patent application identified above.
2. I understand that the Examiner has cited Hei (US 6,024,986) as prior art in prosecution of the application identified above. I understand that the Hei patent was filed on May 24, 1999.
3. I further understand that the filing date of the present patent application Serial No. 09/614,631 is July 12, 2000.
4. I state that before the date of the Hei patent, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above.
5. The claims of the patent application identified above relate to compositions including:

about 35 to about 45 weight-% acetic acid, about 5 to about 15 weight-% octanoic acid, about 3 to about 8 weight-% hydrogen peroxide, about 8 to about

16 weight-% peroxyacetic acid, about 1 to about 5 weight-% peroxyoctanoic acid, and about 0.1 to about 2 weight-% chelating agent;

about 40 weight-% acetic acid, about 10 weight-% octanoic acid, about 5 weight-% hydrogen peroxide, about 12 weight-% peroxyacetic acid, about 3 weight-% peroxyoctanoic acid, and about 0.6 weight-% chelating agent;

about 10 to about 150 ppm acetic acid, about 5 to about 40 ppm octanoic acid, about 4 to about 20 ppm hydrogen peroxide, about 5 to about 50 ppm peroxyacetic acid, about 2 to about 25 ppm peroxyoctanoic acid, and about 0.2 to about 2.5 ppm chelating agent;

about 133 ppm acetic acid, about 33 ppm octanoic acid, about 17 ppm hydrogen peroxide, about 40 ppm peroxyacetic acid, about 10 ppm peroxyoctanoic acid, and about 2 ppm chelating agent;

about 50 to about 60 weight-% acetic acid, about 10 to about 20 weight-% octanoic acid, about 5 to about 15 weight-% hydrogen peroxide, and about 0.3 to about 1 weight-% chelating agent; or

about 54 weight-% acetic acid, about 14 weight-% octanoic acid, about 10 weight-% hydrogen peroxide, and about 0.6 weight-% chelating agent;

Each claimed composition also has at least about 1 part by weight of peroxyoctanoic acid for each about 5 parts of peroxyacetic acid.

The present patent application includes at page 15 several tables describing embodiments of the claimed compositions. Two of the tables describe concentrate and use compositions including:

Chemical	Wt-%	ppm
Acetic Acid	40	133
Hydrogen Peroxide	5	17
HEDP	0.6	2
Octanoic Acid	10	33
Peroxyacetic Acid	12	40
Peroxyoctanoic Acid	3	10

One of the tables describes raw materials that can be used to make the concentrate composition described above. These raw materials include:

Raw Material	Weight %
Glacial Acetic Acid	54
Hydrogen Peroxide, 35%	30
HEDP, 60%	1
Octanoic Acid, 95%	15

6. During the research and development leading to the claimed compositions, and before May 24, 1999, my coinventors and I made several compositions meeting the limitations of the claims of the present application and including the compositions described in the tables in paragraph 5 above.

7. Accompanying Exhibit A reports studies of compositions that meet the limitations of the present claims and including the compositions described in the Tables in paragraph 5 above. In Exhibit A, the initial, concentrate, and use compositions described in the tables in paragraph 5 above are referred to as "Falcon 15 O". The composition called "Falcon 15 AE" also meets the limitations of the present claims. The contents of these formulas can be found at page 9-10 of Exhibit A. Page 9 of Exhibit A describes the stability of these formulas. The pages of the report attached as Exhibit A were prepared before May 24, 1999.

8. The evidence presented in Exhibit A indicates that, before May 24, 1999, my coinventors and I invented the subject matter described and claimed in the patent application identified above. Moreover, Exhibit A demonstrates the concentrations of the compositions called Falcon 15 O and Falcon 15 AE, as invented prior to May 24, 1999, fit within the claims of the present invention. Pages 1 and 9 of exhibit A also demonstrates the compositions of Falcon 15 invented prior to May 24, 1999, were physically stable.

9. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

John D. Hilgren

1-25-06

EXHIBIT A
DATED DECEMBER 23, 2005
APPLICATION NO. 09/614,631

TITLE **F-15 TALKIN IS**

PROJECT NO.

W-299

BOOK NO.

9980

34 That does not require a coupler NAS

Work continued from Page

OBJECTIVE: Develop a concentrated version of Falcon KX-6049
Or develop a sanitizer concentrate that maximizes
actives (POAA, PCOA, H₂O₂) while minimizing costs

Discussion w/ [REDACTED]: [REDACTED] has developed a "Falcon 15" formula that is physically stable.

Formula A)	Glacial Acetic Acid	57.0%	50.87
	H ₂ O ₂ (35%)	25.0	3.50
	Dequest 2010 (60% Active)	1.0	0.60
	Oxalic Acid	15.0	12.28
	POAA	0.0	10.30
	PCOA	0.0	3.02
	Water	0.0	19.07

*The Equilibrium concentrations were based on the "SOLVER" computer program. We chose equilibrium constants based on 3 samples of Falcon 15 produced and analyzed by analytical. $K_1 = 1.7$ & $K_2 = 2.35$

FALCON 15 EQUILIBRIUM CONSTANTS DERNATION

RICHTER/REINHARDT FORMULA

	% Raw Mat'l
Acetic Acid	59.00
Hydrogen Peroxide	8.75
Dequest 2010 (60%)	0.60
Oxalic Acid	15.00
POAA	
PCOA	
Water	16.65
	100.00

Concentration in Molarity

	9807-48	9889-36	9889-48
AAI	10.27	10.27	10.27
H2O2i	2.69	2.69	2.69
OAI	1.09	1.09	1.09
H2Oi	9.66	9.66	9.66
AAeq	8.98	8.04	7.78
H2O2eq	1.02	0.99	1.01
OAeq	0.87	0.78	0.76
POAAeq	1.34	1.43	1.38
PCOAeq	0.30	0.20	0.19
H2Oeq	10.56	14.86	16.16

ANALYTICAL RESULTS

"After Manipulation of SOLVER program we decided:

$K_1 = 1.7$ & $K_2 = 2.35$

2 Week Equilibrium Concentrations (% By Weight)

	9807-48	9889-36	9889-48	SOLVER	SOLVER
	51.57	48.19	44.70	50.87	49.54
	3.33	3.21	3.29	3.50	2.62
	0.60	0.60	0.60	0.60	0.6
	12.00	10.80	10.50	12.28	11.76
	9.73	10.41	10.10	10.30	11.98
	4.57	3.14	2.95	3.02	3.8
	16.20	25.65	27.86	19.57	20.13
	100.00	100.00	100.00	100.24	100.23

Using these K 's.

$K_1 = 2.77$
 $K_2 = 4.00$
Calculated based on analytical



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$K_1 =$	1.54	2.89	2.85
$K_2 =$	3.54	3.95	4.05
K_3 (from eq)	2.80	1.47	1.42

K_1 ave of last two:	2.77
K_2 ave of last two:	4.00
K_3 ave of last two:	1.44

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TITLE Falcon 15

PROJECT NO.

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Work continued from Page 34

According to [redacted], original goal was to maximize octanoic acid content in raw material but they determined a solubility limit near 15%. Physical stability tests are performed at 40°F and 100°F.

It appears from Micro testing that the lower the H₂O, the better. We will focus on (1) Minimizing cost (2) maintain physical stability (3) maintain chemical stability (4) maximize PA/PAH₂O ratios (5) Understand other factors involved that increases efficacy. (6) Make Non-Detectable solutions.

I. Physical Stability TESTS

A) CONCENTRATE

- (1) Room Temp
- (2) 40°F
- (3) 100°F - 1 MONTH?

B) Use Solution - May use

- (1) Room Temp
- (2) 40°F
- (3) 100°F

Analytical G.C. To determine if octanoic separates to the top.

- I. Qualitative - Turbidity
- II. Quantitative - G.C.

II. Chemical Stability TESTS

- concentrate ONLY

- (1) Room Temp
- (2) 100°F

Method of Analysis

- (1) QATM 203
- (3) HPAC

III. EFFICACY

- (1) - At use concentrations
- 4 exposure times / Temp. / Etc.

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FALCON 15

PROJECT NO.

36

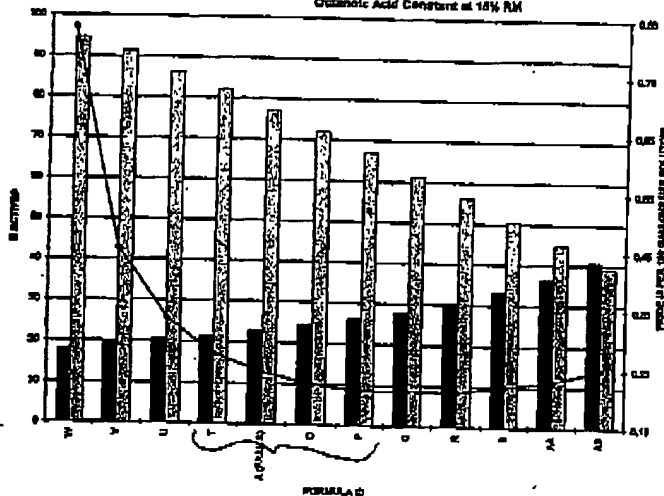
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Important Areas of INTEREST WHEN DECIDING UPON A FALCON 15 FORMULA:

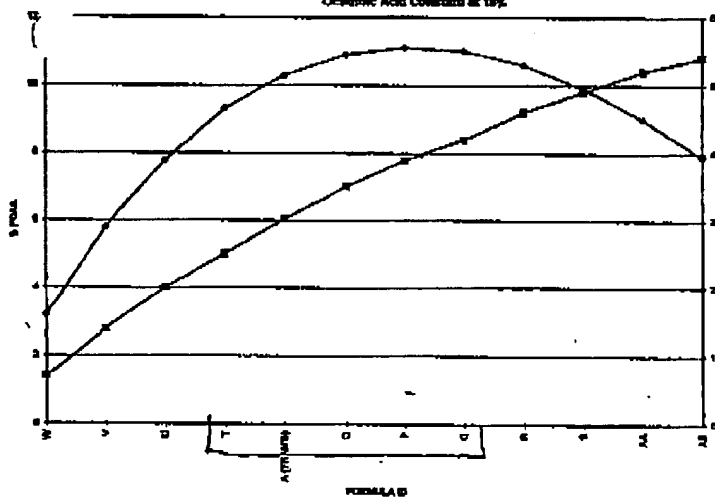
FALCON 16 POTENTIAL FORMULAS
Oxalic Acid Constant at 15% RM



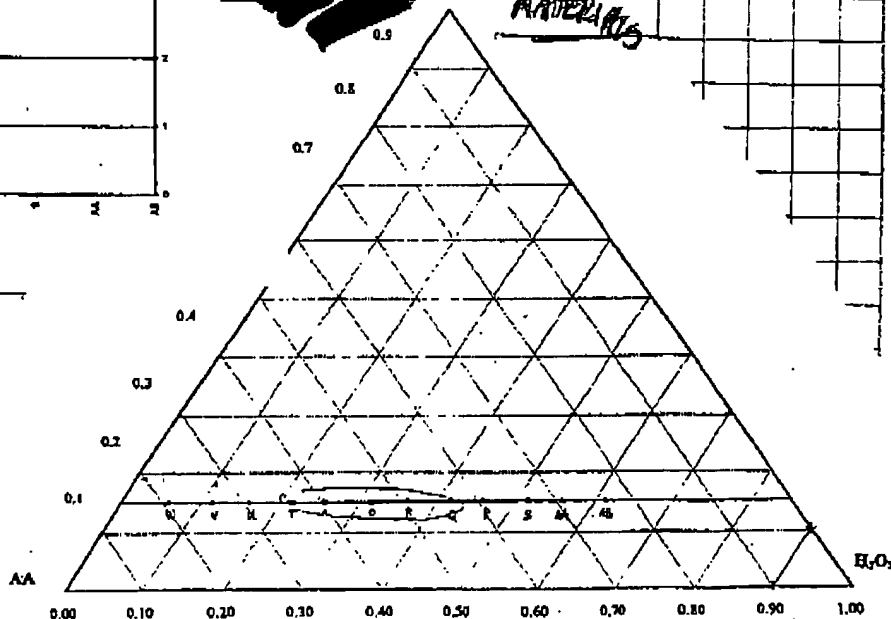
	AA	H2O2
W	75%	0%
V	74%	10%
U	69%	15%
T	54%	20%
A (Perm)	50%	25%
O	34%	30%
P	48%	26%
D	41%	40%
R	30%	45%
S	34%	50%
AA	20%	55%
AB	24%	60%

For Ternary diagram, determine where the detectable formulas are and where the unstable (physically) formulas are.

FALCON 16 POTENTIAL FORMULAS
Oxalic Acid Constant at 15%



RAW MATERIALS



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Work continued from Page **36**

Phys. and Stability TESTS: MIX ALL SAMPLES, 1 LIT/L 15 MINUTE 5 MINUTES - 501 Plate

CONCENTRATION	TEMP.	QUALITATIVE ANALYSIS
Falcon 15 - CONCENTRATE Prepared by Joe Schilling 8/19/96 SG = 1.0443	100°F	START DATE: 12-27-96
102/13 gallons 601 ppm (V/V) Falc = 0.5815 gms 628 (W/W) H ₂ O = 925.8 gms	Room Temp 60°F	MIX w/ Top H ₂ O / A small amount of oil drops seemed to be on the surface but after mixing Note as evident
1202 ppm (V/V) Falc = 1.0175 1235 ppm (W/W) H ₂ O = 810.2	Room Temp 60°F	Once mixed appears stable
1803 (V/V) Falc = 1.8075 1883 (W/W) H ₂ O = 958.4	Room Temp 60°F	An obvious oil separation on top before thoroughly mixing. After mixing: very slight oil droplets.
2232 (V/V) Falc = 1.9020 2331 (W/W) H ₂ O = 815.2	Room Temp 60°F	Oil separation on top before mixing. *obvious oil slick after mixing
6695 (V/V) Falc = 5.610 6992 (W/W) H ₂ O = 798.2	Room Temp 60°F	Heavy oil slick prior to mixing and after mixing
9576 (V/V) Falc = 8.1305 1% (W/W) H ₂ O = 805.1	Room Temp 60°F	Heavy oil slick before and after mixing

SEE PP. 40

* Appears that solubility limit for this Falcon 15 formula is between around 1800 ppm (V/V) at 60°F.

* Could we use a H₂O soluble dye to stand out greater?

$$\frac{10}{(X \text{ ppm})(128)} = X \text{ gallons for 1 oz}$$

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CHEMICAL STABILITY

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BOOK NO.

9980

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Work continued from Page

Objective: Determine if chemical stability changes substantially over time at elevated temperatures.

SOLUTION: [REDACTED] Folcon 15 prepared [REDACTED]

Analytical Results from [REDACTED]:

FORMULA

59% - AA
25% - H₂O (35%) - 8.5% (100%)
15% - OA
1% - Degradant

OA - 8.73%
POAA - 2.67%
H₂O - 4.12%
POAA - 12.30%

LC

Analytical Results

ANALYTICAL

ANALYTICAL RESULTS

Room Temp
Start of Test
COMPONENTS

Room Temp
Start of Test
([REDACTED])

Room Temp
End of 1 month
([REDACTED])

100°F
End of 1 month
([REDACTED])

Acetic Acid

N/A

59.80 50.67

51.22

63.0%

H₂O

4.12%

3.20 3.20

1.50

<0.1%

POAA

2.67%
11.03%

12.20 9.13

7.88

0.40%

POAA

2.67%

2.26 2.26

2.24

0.27%

OA

8.73%

12.20 12.20

13.0

18.1%

8.6% Degradant 0.6% H₂O
11.74% H₂O 21.94% H₂O

OVER START DATE: [REDACTED]

END DATE: [REDACTED]

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PROJECT NO.

BOOK NO.

9980

39

Determine upper OA limit

Work continued from Page

Objective: Prepare the following Falcon 15 formulas to determine physical stability of high OA concentrations.

FORMULA

Room Temp
Stabilizing

40°F
Stabilizing (if Room Temp is stable)

X - 57% - AA - 4.09gms
20% - H₂O₂ - 13.95
20% - OA - 3.90
1% - Dequest - 0.74

STABLE CLEAR
SOLUTION

STABLE CLEAR
SOLUTION

Y - 47% - AA - 34.53gms
30% - H₂O₂ - 21.48
20% - OA - 14.10
1% - Dequest - 0.71

Using 40°F H₂O₂ - When
mixing was not stable,
but once at room
temp, became stable
& clear.

NOT STABLE
Separated into two
layers.

Z - 39% - AA - 34.53gms
40% - H₂O₂ - 21.48
20% - OA - 14.10
1% - Dequest - 0.71

NOT STABLE - Top
Octanoic Acid Layer.

Did not test -
No need to.

* Depending on stability or instability of these formulas we will
vary OA concentration.

Glacial Acetic Acid - 124016 LOT #115371

Octanoic Acid - 170280 LOT #115371

35% H₂O₂ - 240317 LOT #35762-144

QUEST ADPA - 60AW 250282 LOT# 05036-05807P

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Work continued to Page 40

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TITLE 15-10 HADON 15

PROJECT NO.

40

Determine Upper OA Limit.

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9980

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According to "Merck Index" Octanoic Acid has a maximum solubility of 0.068 gms / 100 gms H₂O or (680 ppm w/w). It has greater solubility in acetic acid.

680 ppm (w/w) octanoic acid would be equivalent to

$$\frac{680}{(1.0943)(.1228)} = 5,303 \text{ ppm (v/v) Falcon 15 @ } 25^{\circ}\text{C. or Room Temperature.}$$

S.G. Po octanoic Acid by "solver"

* We noticed solubility problems at 1800 ppm (v/v) although this was at 16°C or 60°F.

* At room temp. we would expect to see slightly better solubility than 5303 due to the coupling ability of acetic acid. Although, we are not sure of the solubility limit of peroxanone acids. - Most likely no limits.

* The samples from pp. 37 were also placed in 40°F refrigerator

	All samples use Falcon 15	STABILITY		
		40°F	60°F	70°F
601 ppm (v/v)		STABLE	STABLE - Actual	STABLE
1202		STABLE	STABLE - Raw	STABLE
1803		STABLE	UNSTABLE - Material	STABLE
2232		STABLE	UNSTABLE - Whole	STABLE
6695		UNSTABLE	UNSTABLE - Samples	UNSTABLE
9576		UNSTABLE	UNSTABLE	UNSTABLE

* NOTE: The 40°F stability tests were performed by sampling from a larger sample while the solution may have already separated. Repeat experiment with large sample.

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TITLE CONTD PS-15

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BOOK NO.

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Additional Concentrations to narrow in on Formulas -
Determining Physical Stability.

FORMULA	ROOM TEMP. STABILITY	40°F STABILITY
Q AA - 44% - 56.79 gmi H ₂ O ₂ - 40% - 51.64 Degumest - 1% - 1.29 OA - 15% - 19.47	NOT STABLE	NOT STABLE
R AA - 39% H ₂ O ₂ - 45% Degumest - 1% OA - 15%	ASSUME INSTABILITY BASED UPON Q & the fact that AA is a coupler.	
Y1 AA - 52% - 54.58 gmi H ₂ O ₂ - 30% - 31.56 Degumest - 1% - 1.15 OA - 17% - 17.86	STABLE	STABLE
Y2 AA - 49% H ₂ O ₂ - 33 Degumest - 1% OA - 17%	{ Assume Instability due to P & Y being unstable - did not prepare this sample.	
Z1 AA - 39% H ₂ O ₂ - 43% Degumest - 1% OA - 17%	{ Assume INSTABILITY due to Q being UNSTABLE	
Z2 AA - 42% H ₂ O ₂ - 40% Degumest - 1% OA - 17%	{ and the fact that AA is a coupler	

RAW MAT'L

PRODUCT LOT #s

* Glacial Acetic Acid: 104016 lot #115371

35% H₂O₂: 240317 lot B5762-144

11-gumest ADPA: 250282 lot 05056-0580DR

Octonox Acid: 170280

lot

219605

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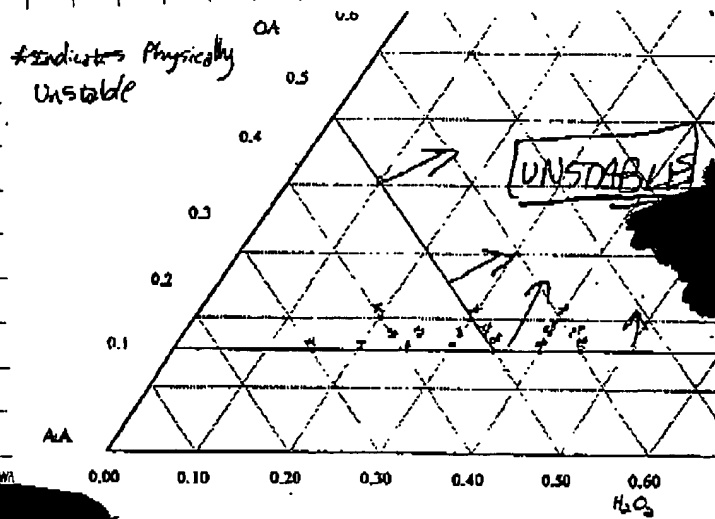
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FORMULA	Room Temp STABILITY	40°F STABILITY
P AA - 49% - 51.65		
H ₂ O ₂ - 35% - 31.0	UNSTABLE	UNSTABLE
Dequest - 1% - 1.05	STABLE	
QA - 15% - 15.81		
X ₂ AA - 57% -	Assume STABILITY Based upon stability of X ₁ , X ₃	
H ₂ O ₂ - 23% -		
Dequest - 1% -		
QA - 17% -		
X ₃ AA - 56% - 31.16	STABLE	STABLE
H ₂ O ₂ - 26% - 21.7		
Dequest - 1% - 1.00		
QA - 17% - 16.24		

*STABILITY CURVE THIS FOR:



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TITLE

CONT'D 45-10

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* Based upon efficacy data from [REDACTED] [REDACTED] [REDACTED] -
lower H_2O_2 proves to be more efficacious.

5	FORMULA	Room Temp. STABILITY	40°F STABILITY
	AC: AA - 49.0 - 36.46gms		
	H_2O_2 - 40.0 - 29.77	STABLE	UNSTABLE
10	Dye - 1.0 - 0.75		
	OA - 10.0 - 7.45		
	AD: AA - 39.0 -	{ Did Not Prepare based upon AC - This will be unstable	
	H_2O_2 - 50.0 -		
	Dye - 1.0 -		
15	OA - 10.0 -		
	AE: AA - 57.0 - 46.04		
	H_2O_2 - 30.0 - 23.44	STABLE	STABLE
	Dye - 1.0 - 0.82		
	OA - 10.0 - 7.81		
20	AF: AA - 69.0 - 48.51		
	H_2O_2 - 20.0 - 14.03	STABLE	STABLE
	Dye - 1.0 - 0.74		
	OA - 10.0 - 7.04		
	A: AA - 59.0 - 39.21		
25	H_2O_2 - 25.0 - 16.63	STABLE	STABLE
	Dye - 1.0 - 0.66		
	OA - 15.0 - 10.00		
	O: AA - 54.0 - 32.57		
	H_2O_2 - 30.0 - 18.11	STABLE	STABLE
30	Dye - 1.0 - 0.62		
	OA - 15.0 - 9.05		
	T: AA - 64.0 - 43.31		
	H_2O_2 - 20.0 - 13.54	STABLE	STABLE
	Dye - 1.0 - 0.69		
	OA - 15.0 - 10.16		

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FORMULAS SENT TO ANALYTICAL FOR ANALYSIS:

FORMULA ID	DATE PREPARED	ANALYTICAL RESULTS	DATE:
		AA - 69% H ₂ O ₂ - 20% OA - 10%	POAA POOA
AF		8.58 58.48 1.79	10.92 1.22
T		11.2 53.28 1.87	10.92 1.45
X		15.8 47.49 1.82	10.11 2.72
A		12.2 48.14 2.98	10.16 2.19
AE		7.45 46.64 3.98	13.46 1.76
O		11.4 40.99 4.15	13.11 2.51

NOTE: AF, T, A, AE & O - Had Equilibration @ room temp for 3 days
X - " " " " " " 27 days

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